Draft Test Plan Vehicle Emissions Tests at ARB's MTA Facility Biodiesel Characterization Study Part One: Fuels

Prepared in collaboration with Tom Durbin

I. Objective

The goal of the proposed study is to conduct emissions tests on three vehicles and three biofuel-derived diesel fuels. The test fuels will include a soy-based and animal-based biodiesel at B20, B50, and B100, a renewable diesel fuel at R20, R50, and R100 and a typical CARB diesel fuel. Emissions from the biodiesel and renewable diesel will be compared to CARB diesel. Testing will include regulated emissions, toxics, greenhouse, and ultrafines. Data obtained from this study will allow comparisons of emissions from vehicles tested on two drive cycles and will allow for health affects assessment. The test fuels and handling and analysis procedures discussed in this document will also be the same as those used for the engine dynamometer testing.

II. Test Fuels

A. CARB Diesel Fuel

A CARB-certified and typical California ultra-low sulfur diesel (ULSD) fuel will serve as the baseline for testing. A CARB ULSD has already been purchased from a California refinery that has specifications similar to the typical specifications listed below. The final specifications for this fuel will be provided after the triplicate analyses have been performed to establish the final fuel properties.

Typical specifications for CARB ULSD in California

- Less than 15 ppm sulfur
- 53 cetane
- 20 percent aromatics
- No cetane enhancers or other additives
- Meets ASTM D975

B. Biodiesel Feedstocks and B100 Fuels

Two biodiesel feedstocks will be utilized for testing, including a soy-based and an animal-based biodiesel fuel. These feedstocks are selected to provide a range of biodiesel characteristics that represent different characteristics of biodiesel in terms of cetane number and degree of saturation. Also, the selection of a soy based vegetable oil and animal based biodiesel may cover a broad range of characteristics that may affect toxic emissions.

- Meets D6751 (see Table 3)
- The feedstocks are BQ9000 compliant
- The feedstocks meet a 6 hour Rancimat for oxidative stability
- An anti-oxidant will be added to the biodiesel fuel
- No anti-microbial will be added to the biodiesel fuels

C. Renewable Diesel Feedstock

A renewable diesel feedstock will also be used for testing. This renewable feedstock will be provided by Neste Oil, and it is known as NExBTL. This fuel is produced from renewable biomass sources such as fatty acids from vegetable oils and animal fats via a biomass to liquid diesel technology.

- Meets ASTM D975
- No cetane enhancers or other additives are added

D. Biodiesel and Renewable Diesel blends

The biodiesel and renewable diesel feedstocks will be blended with the ULSD base fuel in different blending ratios. The soy-based and animal-based biodiesels will be blended at levels of B20, B50, as well as using the straight B100. The renewable diesel fuel will also be blended at levels of R20, R50, as well as using the straight R100.

E. Biodiesel blends

- B5
- Must meet ASTM D975
- Analyze for the properties specified in Table 4 which provides properties and specifications for B20. The B5 must meet these specifications as well as D975 specifications given in Table 2.
- This blend will only be used for the engine dynamometer testing.
- B20
 - Must meet B20 biodiesel blend specifications given in Table 4.
- B50
 - Analyze for properties given in Table 4, however certain limits may not apply.

III. Fuel Analysis

The ULSD and the NExBTL will be tested in duplicate (certificate of analysis will count as the third replicate) upon arrival to the fuel storage facility for all properties under ASTM D975, density (ASTM-D4052), and additional diesel properties as specified in Table 2. The pure biodiesel feedstocks will be tested in triplciate upon arrival to the fuel storage facility for all properties under ASTM

D6751 and for density. The density for the ULSD and the pure biodiesel feedstocks will be utilized for the fuel blending. Preliminary tests will be conducted on the ULSD and the pure biodiesel feedstock prior to shipment/delivery to help ensure these fuels will meet the testing specifications upon arrival.

The biodiesel blends will be tested in triplicate after the fuels have been blended for the B20 fuel properties provided in Table 4 and for ASTM-D7371 for the blend levels.

The renewable diesel blends will be tested in triplicate after the fuels have blended for ASTM D975, other diesel properties, and for density to confirm the blend levels. See Table 2.

The ULSD, the two pure biodiesel feedstocks, and the renewable diesel fuel will all be tested in triplicate for the presence of trace elements, as specified in Table 5.

Table 1. Feedstock analysis

Test Method	CARB	Renewable Diesel	Renewable Diesel Blends	Biodiesel	Biodiesel Blends
D975 + others (see Table 2)	3	3	3	No	No
Density	3	3	3	3	No
Elemental Analysis	3	3	No	3	No
D6751-07b	No	No	No	3	No
B20 properties	No	No	No	No	3
D7371-07	No	No	No	No	3

Table 2. ASTM D975 diesel fuel test method and additional diesel analyses

Property	Test Method
Sulfur Content	D5453-93
Total Aromatic Content	
mass%	D5186-96
PAH mass%	D5186-96
Nitrogen Content	D4629-96
Natural Cetane #	D613-94
Cetane Index	
Gravity, API	D287-82
Viscosity at 40 C	D445-83
Flash Point	D93-80
Distillation	D86-96
ibp (F)	
10% (F)	
50% (F)	
90% (F)	
ep (F)	
Cloud point	D2500
Pour Point	D-97
Ash	D-482
Ramsbottom Residue	D524
Water and Sediment	D1796
Conductivity	D2624
Corrosion	D130

Table 3. D6751 biodiesel feedstock test method

	Property	Limits	Test Method
1	Calcium &	5 max ppm (ug/g)	EN 14538
	Magnesium		
2	Alcohol control	130 °C min or 0.2 vol%	D93 or EN14110
3	Flash Point	93 °C min	D93
4	Kin. Viscosity, 40 °C	1.9-6.0 mm ² /sec	D445
5	Sulfate Ash	0.02 max % mass	D874
6	Sulfur S15	0.0015 max % mass	D5453
		ppm	
7	Copper	No. 3 max	D130
	Corrosion		
8	Cetane number	47 min	D613
9	Cloud Point	Report °C	D2500
10	Carbon Residue	0.05 max % mass	D4530
11	Acid Number	0.50 max mg KOH/g	D664
12	Free Glycerin	.020 % mass	D6854
13	Total glycerin	.240 % mass	D6874
14	Phosphorous	0.001 max % mass	D4951
15	Distillation, T90 AET	360 °C max	D1160
16	Na/K, combined	5 max ppm (ug/g)	EN 14538
17	Oxidation	3 hour min (6 hr min ¹)	EN 14112
	Stability		
18	Visual	Free of un-dissolved	D4176
	Appearance	water, sediment and	
		suspended matter	

^{1. 6} hr min required for this study

Table 4. Biodiesel blend test method EMA test specification for biodiesel fuel (May 31, 2006) Final Blend Fuel Requirements For B20

(IVIA	Property	D2 Blend	Test Method
1	Flash Point, °C, min	52	ASTM D93
2	Water and sediment, vol%, max.	0.05	ASTM D2709 or
			D1796
3	Physical Distillation, T90, °C, max	343	ASTM D86
4	Kinematic Viscosity, cST@40 °C	1.9-4.1	ASTM D445
5	Ash, mass%, max	0.01	ASTM D482
6	Sulfur, ppm, max	15 ppm ¹	ASTM D5453
7	Copper strip corrosion rating, max	No. 3	ASTM D130
8	Cetane Number, min.	43 (48) ¹	ASTM D613
9	Cloud point ²	Per	ASTM D2500
		footnote	
10	Ramsbottom carbon residue on	0.35	STM D524
	10% distillation residue, wt%, ma		
11	Lubricity, HFRR@60°C	460	ASTM D6079
12	Acid number, mg KOH/g, max.	0.3	ASTM D664
13	Phosphorus, wt%, max.	0.001	ASTM D4951
14	Total Glycerin		NA
15	Alkali metals (Na+K), ppm, max.	ND	EN14108
16	Alkaline metals (Mg+CA), ppm,	ND	EN14108
	max		
17	Blend fraction, vol. % ³	± ?	ASTM D7371-07 ¹
18	Thermo-oxidative stability	10	Modified ASTM
	insolubles,		D2274 ⁴
19	Oxidation Stability, Induction time,	6	EN14112
	hours min		(Rancimat)

EMA

- 1. Specification are modified from the EMA recommended requirements to meet the goals of this study
- 2. The maximum cloud point temperature shall be equal to or lower than the tenth percentile minimum ambient temperature in the geographical area and seasonal timeframe as defined by ASTM D975.
- 3. Blend fraction refers to the variation in volume percent of B100 in diesel fuel claimed.
- 4. Use glass fiber filter.

Table 5. Elemental Analysis

Species	Method	Level
Chlorine	TBD	ppm
Ce (cerium)	TBD	ppm
Al	TBD	ppm
Sb	TBD	ppm
Ва	TBD	ppm
В	TBD	ppm
Ca	TBD	ppm
Cr	TBD	ppm
Cu	TBD	ppm
Fe	TBD	ppm
Pb	TBD	ppm
Mg	TBD	ppm
Mn	TBD	ppm
Мо	TBD	ppm
Ni	TBD	ppm
Р	TBD	ppm
Si	TBD	ppm
Ag	TBD	ppm
Na	TBD	ppm
Sn	TBD	ppm
Zn	TBD	ppm
K	TBD	ppm
Sr	TBD	ppm
V	TBD	ppm
Ti	TBD	ppm
Cd	TBD	ppm

IV. Fuel Blending

Blending of the biodiesel fuels will be performed at the fuel storage facility. Fuels will be blended on a gravimetric basis to achieve the appropriate volumetric blend levels. After blending, the biodiesel blends will be tested via method 7371-

07 to ensure the blending was uniform and consistent with the targeted blend values.

Protocol

Blending for the renewable diesel blends will be conducted at the facilities at CE-CERT using a gravimetric method. The finished blends will be tested in triplicate for the properties under ASTM D975.

- Fuels must be properly additized before blending begins. For biodiesel feedstocks anti-oxidants at the recommended dosage will be added to the feedstocks.
- 2. Initial batch will be prepared on a test fuel blend to ensure the protocol works
- 3. Procedure
 - a. Density measurements will be made on representative batches of fuel to ensure uniform density
 - b. Calculate mass and volume blend level percents
 - c. Schematic for blending of fuels
 - d. Equipment and supplies
 - i. pumps
 - ii. transfer lines, fittings
 - iii. Specify materials
 - iv. Totes-polyethylene
 - v. Floor scale
- 4. Safety handling
- 5. Recording of data
 - a. mass measurements
 - b. Tare and total wt
 - c. Temperature
- 6. Blend level determination
 - a. Several blend level samples will be taken at various depths in the Tote to ensure uniform mixing.
- 7. Evaluate result, if successful will mix other fuels
- 8. Post mixing adjustment: adjust blend levels if necessary
- 9. Preparation for storage
 - a. Transfer to drum and provide nitrogen blanket
 - b. Schematic for nitrogen setup
- 10. Labeling of drums-provide labels that do not fade due to exposure to fuels 11. QA/QC
 - Tracking and logging of fuels to ensure proper fuels are used for making the fuels
 - b. Record weights and volumes
 - c. Calibration of scale
- 12. Inventory of fuels

V. Fuel Storage

Short term storage

Innerstate Woodland facility in a non-temperature and non-humidity controlled building. The fuels shall be stored under nitrogen and in drums.

Long term storage

CeCERT and another location TBD

Limited quantities of the fuels will be stored on-site at the Stockton and MTA test facilities.